

## A Five-Year Cold Storage Experiment with Salted Calfskins†\*

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The cold storage of hides and skins and especially calfskins has been practiced for some time by a number of tanners. Nevertheless, there are today practically no data available on the length of time that green salted hides and skins may be cold-stored without adversely affecting the quality of leather made from them. The economic importance of such information may well be illustrated by the experiences of the Government of the United States during the drought of 1934 when it was faced with the problem of holding several million hides and skins resulting from the cattle killing program.

It was recognized then that much of this raw stock probably would have to be carried for exceptionally long periods, measured perhaps in years rather than in months. In view of the foregoing possibility and the lack of actual data, it was decided after much deliberation that cold storage offered the most logical opportunity of carrying out this program with a minimum of deterioration or spoilage of raw stock. There can be little doubt but that the adoption of this procedure played an important part in the highly successful completion of this undertaking, as will be evidenced by the data and observations that follow.

Through the cooperation of the Federal Surplus Commodities Corporation a lot of calfskins was made available in 1934 for a long-time cold storage experiment. These skins came from the Midwest. They were classified as 8-12 lb. packer, No. 1 selection, unbranded calfskins, of September take-off. The pack in which they were cured was started on September 6, 1934 and closed October 2, 1934. The skins were cured with all new vacuum pan granulated salt, a point to be emphasized. During the first week of February, 1935 they were bundled and shipped from Oklahoma City, Oklahoma to Baltimore, Maryland and placed in cold storage until March, 1936. The temperature during this period of storage ranged from 32° to 38° F.

In March, 1936 the lot of skins was transferred to a cold storage room in the Bureau of Agricultural Chemistry and Engineering at Washington, D. C. for continuation of the experiments. All of the skins in the lot were

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distinctly of a mature type. A selection was made of 192 skins that most closely resembled typical calfskins. Even so, the majority of them were of the "buttermilk" or "grasser" class.

The 192 selected skins were divided, without discrimination, into four lots of 48 skins each. Although of western origin, the skins when received had a close trim, with the head cut off and the shanks removed at the knees. They were retrimmed to a slight extent by removing occasional ragged edges and parts of udders. The skins in each lot were permanently numbered from 1 to 48 by punch holes in the butt flap on each side of the backbone line.

Each skin was split carefully down the backbone line into practically equal sides and the weight of each side was recorded. By lot the sides were placed in two separate piles of alternating right and left sides, thus giving two half-lots made up entirely of opposite sides of the same skins. Each half-lot was then permanently identified by a system of punch holes in the neck on each side of the backbone line.

Each half-lot was bundled, six sides to the bundle. In doing this the sides were taken in order as piled so that those of the same number in each lot occupied corresponding positions in the bundles.

The bundles were piled in the cold storage room on a platform two inches from the floor. They were put down in accordance with the order in which they were to be removed for tanning. Those in lot 4 constituted the bottom layer, lot 3 came next, followed by lot 2. The first half of lot 1, representing sides cold-stored for one year, was set aside for tanning. The other half of this lot was piled as the top layer. During storage the entire pile was kept covered tightly with a doubled, heavy khaki tarpaulin to reduce evaporation. At yearly intervals each bundle was weighed. Those scheduled for tanning were removed at the time and the remaining bundles were repiled as described. In addition to the period of one year in Baltimore, cold storage was continued in Washington for four years.

The temperature during the four-year period ranged from 30° to 44° F. These extremes were not frequent or prolonged. The mean daily temperature was 36° F. with a deviation of  $\pm 1.5^\circ$  F. There was no permanent installation for humidifying the atmosphere or circulating the air in the cold storage room and, consequently, there were wide fluctuations in relative humidity. To offset this condition somewhat the floor of the room was kept wet most of the time. The daily average relative humidity was 71 per cent with a deviation of  $\pm 10$  per cent.

Changes in weight during the cold storage of the bundled and piled sides are given in Table I. The losses are referred to the bundled weight of the trimmed sides at the time that they were placed in cold storage at Washington, D. C. in 1936, after prior cold storage in Baltimore, Maryland, for one year.

The schedule by which the sides were removed from cold storage and tanned was as follows:

Lot No.	Lot Half	Cold-Stored	Tanned In
1	1st	1 year	1936
1	2nd	2 years	1937
2	1st	2 years	1937
2	2nd	3 years	1938
3	1st	3 years	1938
3	2nd	4 years	1939
4	1st	4 years	1939
4	2nd	5 years	1940

The loss in weight by the green-salted sides during the cold storage at Washington was 3.5 per cent after one year; 5.6 per cent after two years; 9.2 per cent after three years; and 19.2 per cent after four years. The comparatively high loss for the last year was due in part to lower prevailing relative humidities in the cold storage room during this period. Excessive loss in weight of green-salted stock during cold storage presents quite a problem. Laboratory studies<sup>1</sup> completed while these experiments were under way indicate that extremely high relative humidities of from 90 to 95 per cent are necessary to prevent significant losses in weight during the storage of salt-cured calfskins.

TABLE I  
LOSS IN WEIGHT DURING COLD STORAGE  
After Cold Storage at 36° F. ± 1.5° F. for:

Lot No.	Lot Half	Bundled <sup>1</sup> Weight When Stored Lbs.	One Year			Two Years			Three Years			Four Years		
			Weight Lbs.	Loss Lbs.	Per cent	Weight Lbs.	Loss Lbs.	Per cent	Weight Lbs.	Loss Lbs.	Per cent	Weight Lbs.	Loss Lbs.	Per cent
1	1st	233.7												
1	2nd	231.5	219.4	12.1	5.2									
2	1st	230.5	222.2	8.3	3.6									
2	2nd	230.0	225.3	4.7	2.0	220.3	9.7	4.2						
3	1st	231.7	226.9	4.8	2.1	222.0	9.7	4.2						
3	2nd	230.2	223.4	6.8	3.0	218.0	12.2	5.3	212.0	18.2	7.9			
4	1st	229.5	220.6	8.9	3.9	214.8	14.7	6.4	210.1	19.4	8.5			
4	2nd	231.6	220.9	10.7	4.6	213.4	18.2	7.9	205.8	25.8	11.1	194.3	37.3	19.2
Average				3.5			5.6			9.2			19.2	

<sup>1</sup>Weight of retrimmed, bundled sides when placed in cold storage in Washington, D. C., after cold storage in Baltimore, Maryland for 1 year.

Throughout the experiments all sides were tanned at the same tannery. Special efforts were made to maintain the entire process of tanning and finishing constant for all lots. The tannage was a typical American one-bath chrome process for calfskin shoe-upper leather. The sides were given a semi-aniline finish so that defects would not be masked. Some of the significant tannery records and data on yields are given by half-lots in Tables II and III.

TABLE II  
TANNERY WEIGHT RECORDS BY HALF-LOTS

History of Sides	Lot 1		Lot 2		Lot 3		Lot 4	
	1st Half Tanned in 1936 Lbs.	2nd Half Tanned in 1937 Lbs.	1st Half Tanned in 1937 Lbs.	2nd Half Tanned in 1938 Lbs.	1st Half Tanned in 1938 Lbs.	2nd Half Tanned in 1939 Lbs.	1st Half Tanned in 1939 Lbs.	2nd Half Tanned in 1940 Lbs.
Before soaking.....	234	216	220	220	221	212	209	194
After pickling <sup>1</sup> .....	210	208	211	180	195	193	195	210
After tanning <sup>2</sup> .....	221	210	220	225	235	225	220	170
After shaving.....	148	140	148	145	140	142	150	158
As finished leather <sup>3</sup> ....	58	58	58	58	57	59	60	59

<sup>1</sup>After horsing and draining from 24 to 48 hours.

<sup>2</sup>After horsing and draining 24 hours.

<sup>3</sup>After conditioning at 70° F. and 50 per cent relative humidity.

TABLE III  
LEATHER YIELDS BY WEIGHT AND AREA

Lot		Salted Sides in Cold Storage Years	Net Weight <sup>1</sup> of Sides When Stored Lbs.	Weight <sup>2</sup> of Finished Leather Lbs.	Area of Finished Leather Sq. Ft.	Leather Yield	
						On Weight of Stored Sides Per cent	Area per Lb. of Stored Skin Sq. Ft.
1	1st	1	232	58.1	353.3	25.0	1.52
1	2nd	2	231	57.6	344.3	24.9	1.49
2	1st	2	230	58.0	335.3	25.2	1.46
2	2nd	3	230	58.3	354.0	25.3	1.54
3	1st	3	231	56.9	349.8	24.6	1.51
3	2nd	4	230	59.0	340.3	25.6	1.49
4	1st	4	230	60.0	348.0	26.1	1.52
4	2nd	5	231	58.8	337.8	25.5	1.46

<sup>1</sup>Weight of retrimmed sides prior to bundling and placing in cold storage in Washington, D. C.

<sup>2</sup>Weight of finished leather after conditioning at 70° F. and 50 per cent relative humidity.

From the close agreement of the figures in Tables II and III for one year as compared to another it is evident that at least there was no spoilage or deterioration over the entire period sufficient to be reflected in either the behavior of the raw stock in process or in the weight and area yields of finished leather.

As soon as the leather was tanned it was sorted and graded at the tannery. This was done throughout the four-year period by the same personnel. After grading, the leather was shipped to the laboratory where the first half of each lot was held for one year and then returned to the tannery for direct comparison with the leather made from opposite sides of the same skins that had been cold-stored an additional year.

Grading was done on a numerical scale from 1 to 7, in which grade 1 represented the finest leather. Each grade was selected for weight using the gradations of L—LM—PLM—M—HM, in which L was for light weight, M for medium, P for plump, and H for heavy. All of the leathers fell in the low grades of 5, 6, and 7. This was due almost invariably to defects that were clearly of ante mortem origin, such as skin diseases, scratches,

cuts, ribs, and so on. They were not of a character that could be attributed to damages that might have developed during curing, storage, and processing. The true significance of the data on grades and selection so far as the influence of storage is concerned lies in the direct comparison of mated sides from the same skin. The results on grades and selections by half-lots are given in Table IV.

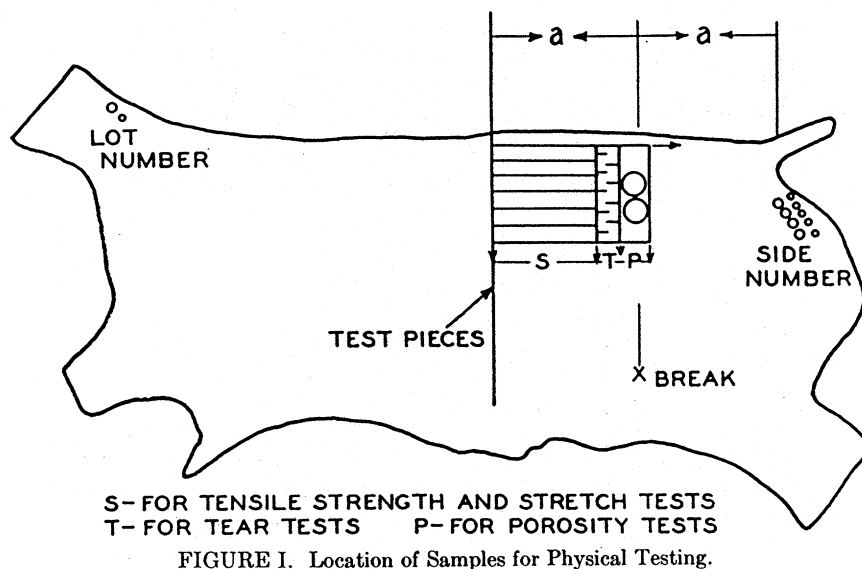
TABLE IV  
GRADES AND SELECTIONS OF LEATHER BY HALF-LOTS

Lot		Tanned in	Grade 4				Grade 5				Grade 6				Grade 7			
No.	Half		LM	M	PM	Total	LM	M	PM	Total	LM	M	PM	Total	LM	M	PM	Total
1	1st	1936	0	1	0	1	0	2	3	5	1	7	9	17	0	9	16	25
1	2nd	1937	0	0	0	0	0	4	2	6	1	16	12	29	0	6	7	13
2	1st	1937	0	1	0	1	0	3	5	8	1	5	12	18	1	11	9	21
2	2nd	1938	0	0	1	1	0	5	4	9	2	10	8	20	2	10	6	18
3	1st	1938	0	1	0	1	0	8	4	12	1	12	5	18	4	10	3	17
3	2nd	1939	0	0	0	0	0	8	4	12	3	9	9	21	2	9	4	15
4	1st	1939	0	1	2	3	1	9	4	14	1	11	7	19	0	6	6	12
4	2nd	1940	0	0	1	1	1	8	4	13	1	15	9	25	0	4	5	9

A summation of the grading data in Table IV shows no lowering of grades with an increase in the length of time that the salted sides were held in cold storage. This was quite in accordance also with the observations by the tannery personnel on the general character of the leather. In no case was the leather in any second half of a lot considered lower in quality than that made from the first half of the lot.

From each half-lot of leather every fourth side, starting with number 1, was selected for sampling for physical tests and analysis. All pieces for physical testing were taken from exactly corresponding locations in each side, as illustrated in Figure I. A perpendicular was dropped from the backbone line through the center of the rear break. A second perpendicular was dropped at a distance equal to that of the first one from the root of the tail. Starting at the location of the second perpendicular, a piece 9 inches long by 6 inches wide and from 0.5 to 1 inch in from the backbone line, was marked off for physical tests. Six pieces for tensile strength and stretch, five squares for tear resistance, and two discs for porosity measurements were cut with dies from each selected side, as shown in Figure I.

All samples were conditioned and tested at 70° F. and 50 per cent relative humidity. Tensile strength and stretch tests were made with a manually operated Schopper tester, supplemented by the use of a vertical electrically operated Scott tester in a few cases in which the breaking load exceeded 100 kilograms. Tear measurements were made with a small hydraulic Schopper tensile strength tester having a capacity of 50 kilograms. Porosity to air was determined with a flow meter under a vacuum of 40 cm. of mercury. The results of these tests are given as averages for each half-lot in Table V. The figures for tensile strength, stretch, tear resistance, and



porosity are averages, respectively, of 72, 72, 60, and 24 individual tests. Here again the concordance throughout the entire experiment in the physical properties listed in Table V shows that no significant changes occurred during the five-year storage period in the leather-making properties of the raw stock.

TABLE V  
AVERAGE TENSILE STRENGTH, STRETCH, TEAR RESISTANCE, AND POROSITY  
OF LEATHER BY HALF-LOTS

Lot. No.	1		2		3		4	
Lot Half	1st	2nd	1st	2nd	1st	2nd	1st	2nd
From Salted Sides Cold-Stored	1 year	2 years	2 years	3 years	3 years	4 years	4 years	5 years
Tensile strength, lbs. per sq. in.....	7,300	7,864	7,590	7,740	7,951	7,878	8,962	8,388
Stretch at breaking load, per cent..	30	32	30	32	33	32	30	29
Tear resistance, lbs.....	11	11	12	11	12	11	12	10
Porosity, mls. <sup>1</sup> .....	222	183	207	172	168	191	171	186

<sup>1</sup>Expressed as mls. of air passed per minute through 1.267 sq. cm. of leather under a vacuum of 40 cm. of mercury.

Using equal weights of the remnants from the pieces cut from each selected side for physical tests, a composite sample representing each half-lot was prepared for chemical analysis, the results of which are given in Table VI. Like the data already presented on yields, grades, and physical properties, those on chemical composition are in splendid agreement throughout and, consequently, are also indicative of no appreciable changes in the condition of the cured sides during cold storage. Indeed, these figures constitute a striking example of the excellent regularity that is possible in the production of chrome leather under experienced and chemical control.

TABLE VI  
CHEMICAL ANALYSIS OF THE FINISHED LEATHER  
(Results expressed on moisture-free basis)

Lot No.	1		2		3		4	
Lot Half	1st	2nd	1st	2nd	1st	2nd	1st	2nd
From Salted Sides Cold-Stored	1 year	2 years	2 years	3 years	3 years	4 years	4 years	5 years
Petroleum ether extract, per cent. . .	2.5	3.3	2.8	3.2	3.3	3.3	3.4	3.4
Total ash, per cent. . . . .	4.3	4.4	4.2	4.2	4.2	4.5	4.3	4.2
Total Cr <sub>2</sub> O <sub>3</sub> , per cent. . . . .	3.5	3.7	3.5	3.6	3.6	3.7	3.8	3.4
Hide substance, per cent. . . . .	88.0	86.7	88.4	85.4	87.1	86.2	86.3	87.4
Combined acid sulfate, per cent. . . .	2.7	2.8	2.6	2.7	2.8	2.8	2.9	2.6
Basicity, per cent. . . . .	51.4	51.7	51.9	52.1	52.2	52.2	52.0	52.2
pH <sup>1</sup> . . . . .	3.38	3.40	3.29	3.35	3.37	3.28	3.30	3.36

<sup>1</sup>On basis of air-dried leather.

### Summary

These experiments conducted on a semicommercial scale provide strong evidence that well-cured, green-salted calfskins may be cold-stored over a period of at least five years without suffering significant changes in their leather-making qualities. This conclusion is supported by strictly comparable quantitative data that show no significant differences in yields, grades, selections, physical properties, and chemical composition of the leather made under rigid control from mated calfskin sides taken from cold storage at yearly intervals.

The skins used in these experiments were well cured with all new, clean salt. They were in an excellent state of preservation when placed in cold storage. It is conceivable that the outcome of such prolonged storage, especially as regards permanent stains and grain damage, might well have been otherwise if old, dirty, bloody salt had been employed in part or entirely for curing, or if the skins had shown evidence of appreciable microbial activity. The results presented herein should not be construed as evidence that improperly cured skins, or those in an advanced state of deterioration, could be stored with safety over an equally long period of time.

Incidental to the major objective of these studies, the data are of value for research purposes in showing the excellent concordance that it is possible to obtain in experimental work carried out in the tannery over a prolonged period.

### REFERENCE

1. Some Aspects of Humidity Control in the Storage of Salt-Cured Calfskins. By L. S. Stuart and R. W. Frey, *J.A.L.C.A.*, 35, 432 (1940).

### Discussion

F. L. DEBEUKELAER: I do not think it is necessary for me to emphasize that this is a very important piece of work for both the packer, the producer of cured skins and the tanner. It is certainly gratifying to know that

properly cured hides evidently can be held for five years' storage, under proper conditions of course.

I wish to emphasize the point that Dr. Frey has mentioned, in regard to the use of proper salt. I think there is no case of packer-cured skins but what 100 per cent new salt is used on them. Of course, unless we do that, they certainly would have salt stains and other defects after a prolonged period, even at low temperature.

This test is also interesting in another direction, in that it will be a forerunner, and something with which to compare the results of a similar test that is being conducted on brined hides for the same period of five years. This test is being run in cooperation with the Warehousing Committee of the Commodity Exchange, Inc. for the purpose of certificating brined hides. It will be very interesting to see what the results of those are in comparison with these calf-skins.

There is, of course, another angle that would enter into the keeping quality of skins, no matter how much care is taken in the use of new salt and proper storage and so on, which we are all aware of. That is the type of bacterial flora contamination. We might have cases where there would be a contamination of flora that could thrive at the low temperatures. We might in those cases have some damage, but that is probably very remote.

I have no specific questions to ask Dr. Frey. He certainly has conducted a very careful experiment here and has tied up very well with the tannery. It is encouraging to see that there is that close cooperation between the research men and the industry.

A. C. ORTHMANN: Mr. Frey, were these in piles or bundles—these sides?

FREY: In bundles. We had cut them into sides, so we bundled six sides to a bundle.

H. B. WALKER: In the physical tests that were made on the finished leathers from these skins, in the case of the tensile strength, far from being deterioration, there was an actual increase in the tensile strength of 12 or 14 per cent, from the first year to the final year. I would like to ask Dr. Frey if they considered that increase significant, or simply accidental.

R. W. FREY: The increase was particularly in the last two lots, and they were the oldest skins and the driest. We wondered if that was the connection. We do not know. We checked the results, and it is possible that the fact that they were the driest skins might in some way account for the high increase in tensile strength.

ORTHMANN: I wonder if we could not tie that up with what Mr. Roddy told us about the coagulable albumens, we probably had in that case a thorough removal or solubilization of them, and they were thoroughly removed in the soaking and washing process.

FREY: I don't know whether you can tie it up or not; but I thought, when he made that remark a while ago, that it might be possible.